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BRAYTON CYCLE CAVITY RECEIVER DEVELOPMENT

QUARTERLY REPORT

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TRW ELECTROMECHANICAL DIVISION

THOMPSON RAMO WOOLDRIDGE INC.
23555 EUCLID AVENUE ■ CLEVELAND, OHIO 44117

JANUARY 1964 - MARCH 1964

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1: **BRAYTON CYCLE CAVITY RECEIVER DEVELOPMENT**

→ **QUARTERLY REPORT, Jan. - Mar. 1964**

(NASA Contract NAS 3-2779)

**Technical Management
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JANUARY 1964 - MARCH 1964

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1.0 PROJECT OBJECTIVES

The Brayton cycle cavity receiver development program as presently planned consists of three phases. Phase I is being performed currently and features both a design study of the full-scale flightweight unit and a material compatibility investigation with lithium fluoride as the corrosive salt. Phase II is contemplated to consist of construction and ground test of the flightweight unit. Phase III is planned as the endurance test of the flightweight unit. The ultimate objective of the program is to demonstrate a one year endurance capability of the flightweight unit in a ground test.

2.0 PROJECT OBJECTIVES FOR THE REPORTING PERIOD OF JANUARY 1, 1964 THROUGH MARCH 31, 1964 (REPEATED FROM THE PREVIOUS QUARTERLY REPORT)

During the quarter from January through March, effort will be directed towards accomplishing these tasks:

1. Completion of the full scale flightweight concept layout.
2. Completion of the small-scale experiments, including data reduction and analysis.
3. Completion of the cavity surface temperature control and aperture closure study.
4. Completion of the design comparisons for the four conditions specified.
5. Completion of the reliability studies for Phase I.
6. Continuation of the first 2500-hour furnace test.
7. Preparation of the rough draft for the topical report on the design study.

3.0 PROJECT PROGRESS DURING THE REPORTING PERIOD

3.1 Task I - The Design Study

3.1.1 Overall Progress

The project schedule for Task I is presented as Figure 1. It can be observed from the schedule that all technical effort on this task was to be completed by 1 March 1964. The technical effort defined in the work statement was completed on schedule. A major technical presentation was made to NASA technical personnel on March 5 and 6. As a result of this presentation, NASA technical personnel decided they need additional information prior to making a firm decision.

Preliminary discussions about the added work indicate that it will probably fall into three categories:

1. Small scale experiments - additional testing and analysis are needed to confirm the previous test results and provide a better analytical basis for understanding the heat transfer process from a hot surface to a storage bath by radiation and/or conduction through extended surfaces, under transient cyclic conditions.
2. Flight type design - a new design is desired utilizing two (2) percent of the heater inlet pressure as the allowable gas pressure drop. The present design employs four (4) percent for the allowable pressure drop. This change is desired to allow a margin of safety for the rotating machinery performance which may not achieve the lofty levels required. Cavity surface temperature distributions are to be determined for the new design. These calculations will employ the collector characteristics predicted by the design studies conducted under contracts NAS 3-2782 and NAS 3-2789. Actual hardware design is to be conducted on the most promising cavity surface temperature control and aperture closure concept. The design study to date has investigated several concepts and arrived at one which appears best for this application.
3. System coordination - additional effort is needed to afford a suitable method for mounting the cavity receiver into the system package and for design evaluation of several suggestions proposed by NASA at about the time of the major technical presentations. These suggestions, if adopted, would result in lower stress levels and presumably improved reliability.

It is anticipated that funding for the additional effort will be made available by NASA on or about 1 June 1964 and that about four (4) months will be required for the design effort.

PROJECT TITLE: Brayton Cycle Lithium Fluoride Cavity Receiver Design Study

DATE: 24 July 1963

ITEM	MONTH OF	JULY	AUGUST	SEPT	OCTOBER	NOV	DEC	JAN '64	FEBRUARY	MARCH	APRIL	MAY	JUNE
1. Preliminary Design Analysis													
2. Lithium Fluoride Properties Investigation													
3. Small Scale Thermal Resistance Experiments													
4. Cavity Temperature Control Study													
5. Fabrication Process Study													
6. Stress Analysis													
7. Flightweight Concept Layout													
8. Reliability Studies													
9. Topical Report													
REPORTS AND PRESENTATIONS													
1. Monthly Reports													
2. Preliminary Design Concept Presentation													
3. Quarterly Reports													
4. Proposal for Phase II Effort													
5. Flightweight Design Concept Presentation													
6. Design Study Topical Report													

FIGURE 1

3.1.2 Preliminary Design Analysis

The completed preliminary design analysis effort was described in the second quarterly progress report, ER-5736, and will not be repeated here. The results indicated that three cavity receiver shapes were possible:

1. Hemisphere
2. Cone
3. Cylinder

Potential concepts illustrating each of these shapes were shown in ER-5736. These various concepts were evaluated in the full-scale flight design effort discussed later, and an optimum design was chosen for completion.

Since the results of the small scale experiments were necessarily applied to the full scale flight design, these results will be presented prior to the flight design discussion.

3.1.3 Small Scale Experiments

The small-scale experiments conducted on this project are intended to determine:

1. The thermal conductivity of lithium fluoride in the liquid and solid states near the melting point.
2. The heat input possible to lithium fluoride containers with several forms of extended surface.
3. The heat release characteristics of lithium fluoride to a gas coolant.
4. Insight into the melting and freezing characteristics of lithium fluoride and the resultant void formation.

The various modules constructed and tested in this program are listed below:

<u>Module No.</u>	<u>Type</u>	<u>Material</u>	<u>Test Purpose</u>
1	Plain	HS 25	Thermal Conductivity
2	Plain	HS 25	Heat Input
3	Plain	HS 25	Heat Release
4	1/16 in. Fins	316 SS	Heat Input
5	1/8 in. Fins	316 SS	Heat Input
6	3/4 in. Honey	316 SS	Heat Input
7	3/8 in. Honey	316 SS	Heat Input
8	Plain, Narrow Annulus	316 SS	Heat Release

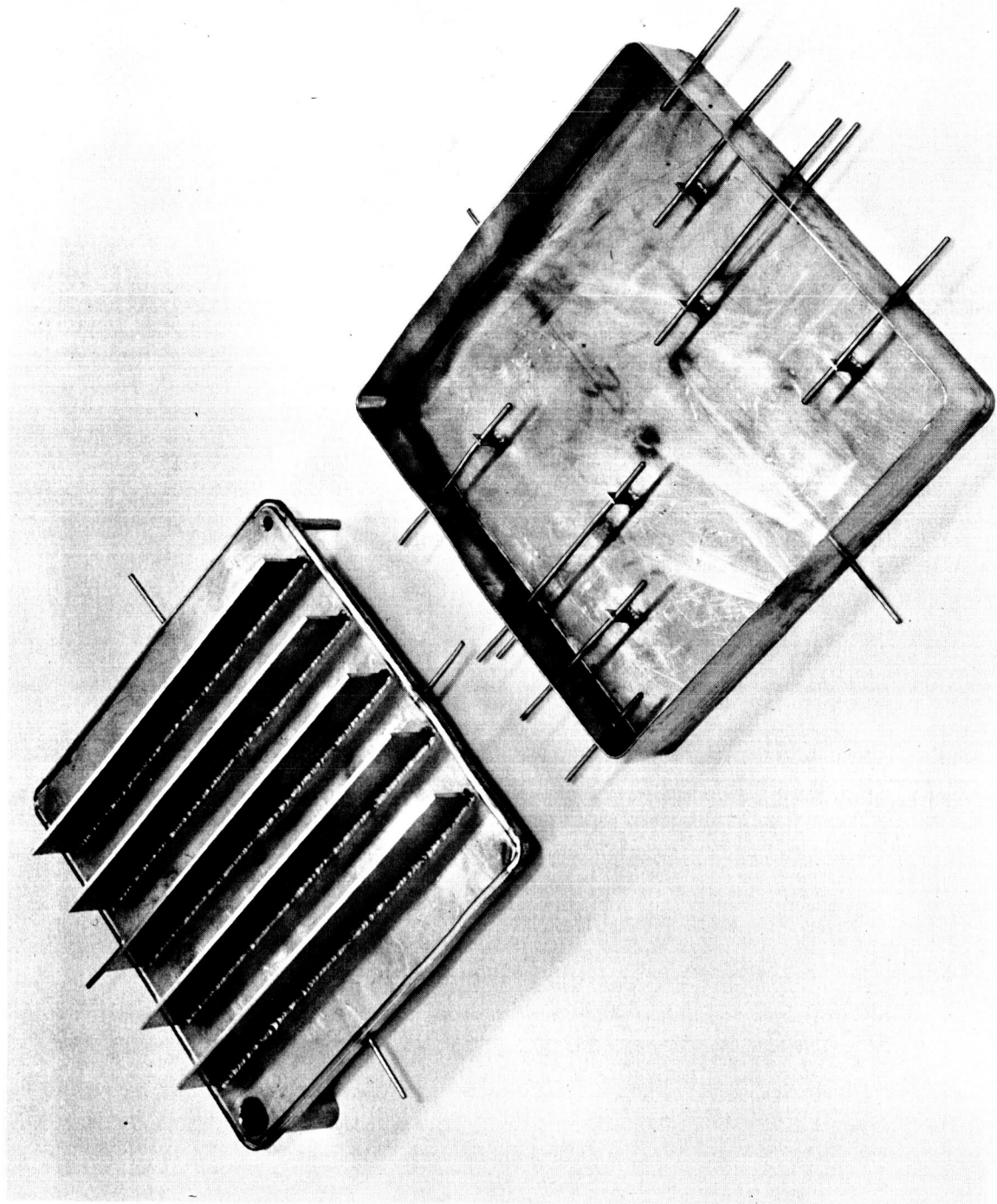
A photograph of module number 5 just prior to the final assembly weld is shown in Figure 2. A similar photograph showing the top half of module number 6 is given in Figure 3. Figure 4 is the same type of shot for module number 7. The tubes shown in all of the above photographs are thermocouple wells, in which 1/16 in. shielded alumel-chromel thermocouples were placed.

An isometric cutaway view of the test bed is presented in Figure 5. A top view prior to filling the enclosure with perlite is shown in Figure 6. The heater leads are uncovered at one end and the thermocouple leads can be readily observed. The 1/4 in. O.D. argon bleed tube to the reflector and the 3/4 in. O.D. fill tube are plainly visible. Module number 1 is installed in the test fixture in Figure 6.

The thermal conductivities were determined in steady-state tests. The heater power setting was maintained constant and the coolant flow adjusted until the bath temperatures remained constant within one degree for approximately one hour. With the bath temperatures constant over such a period, the data obtained over that period was considered valid for reduction. Particular care was taken to make sure that the lithium fluoride was all melted for the liquid case and all frozen for the solid case. In addition, the heat input rate was adjusted for each case to provide as large a temperature difference as possible between the two bath levels at which the temperature is measured. The purpose of the large temperature difference was to reduce the effect of logger reading error. This largest possible temperature difference was important because the test fixture had been sized for a thermal conductivity much lower than actually encountered. Thus, the actual measured temperature difference was much less than had been anticipated.

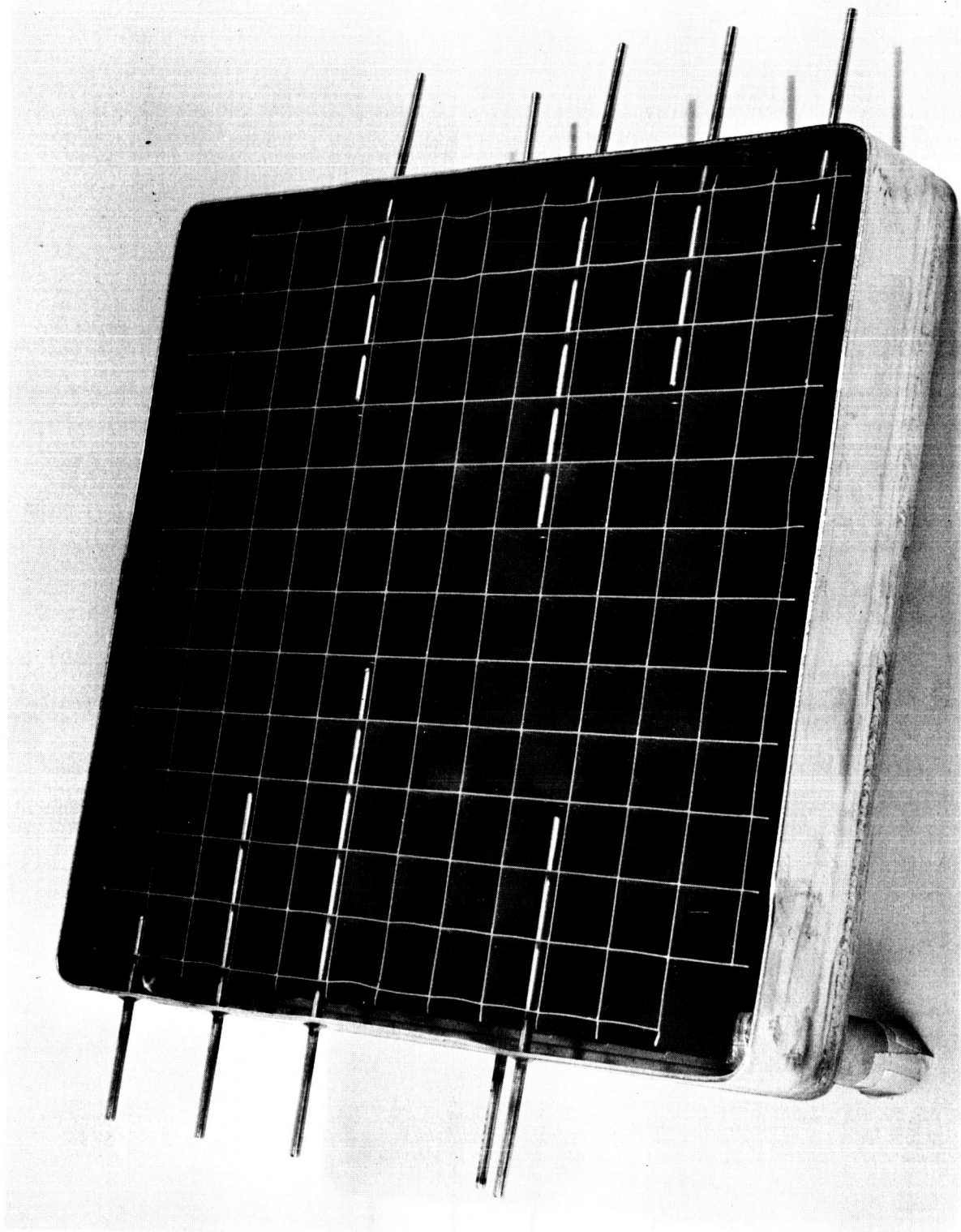
Typical temperature profiles are shown in Figure 7 for the liquid case and in Figure 8 for the solid case. An independent check of the temperature gradients which were employed in the calculation of the thermal conductivities was performed by a senior engineering specialist. He confirmed that the thermal gradients were acceptable. Unfortunately, the other half of the information needed to calculate the thermal conductivities, the heat flux, has not been confirmed by an independent appraisal. Thus, the values obtained are tentative only, and the data will be subjected to a continuing review to substantiate the heat flux calculation. It is preferred not to publish the data at this time until all elements have been checked and confirmed.

The heat input and heat release tests were transient in nature, of course. In practice, the lithium fluoride was melted completely to serve as the reference condition. The coolant flow was held constant to freeze all of the lithium fluoride on each cycle. The cycles went from the all melted condition to the all frozen condition and back to the all melted.



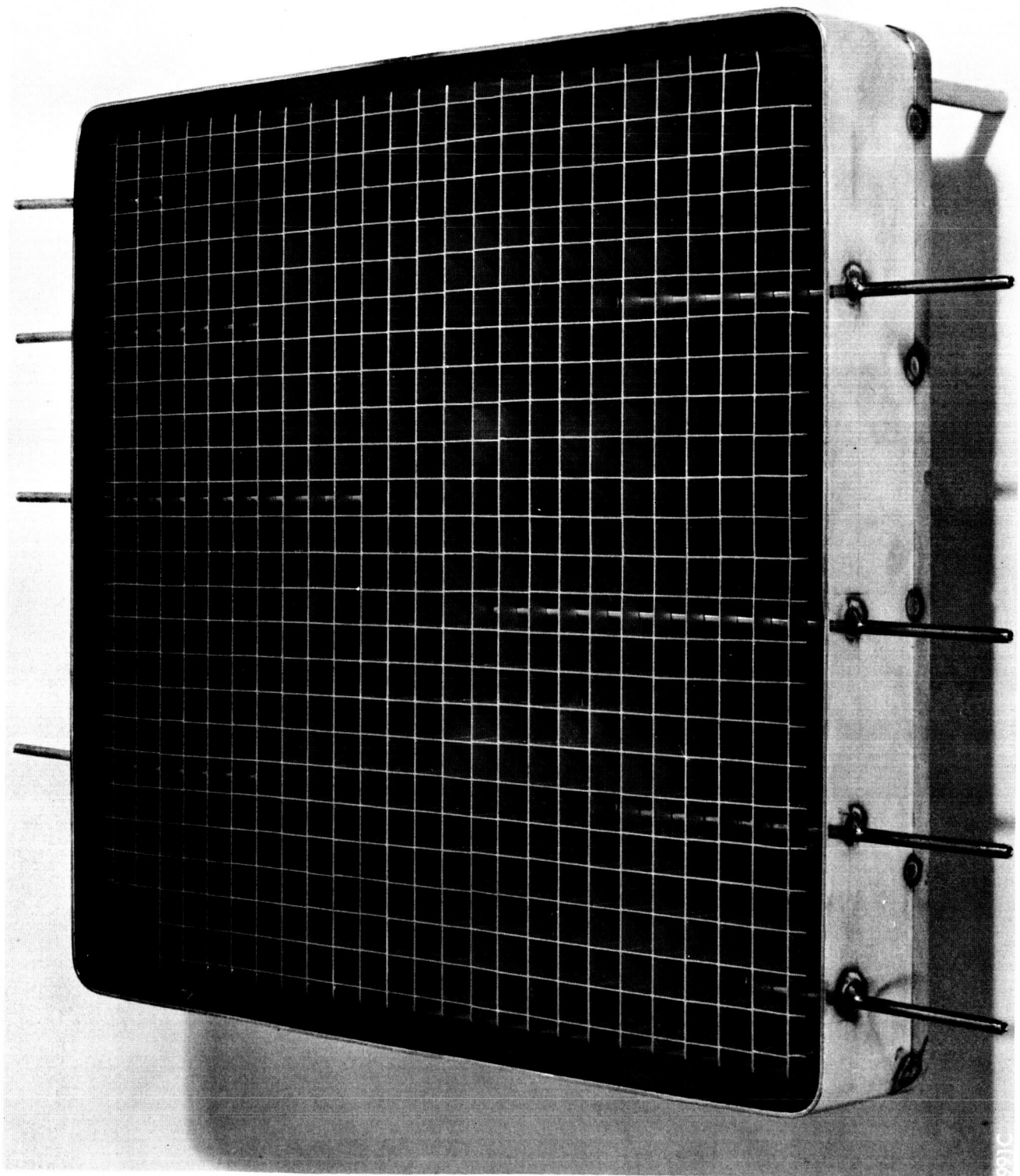
EXPLODED VIEW OF MODULE NO. 5 PRIOR
TO FINAL ASSEMBLY WELD

FIGURE 2



END VIEW OF MODULE NO. 6 PRIOR TO FINAL
ASSEMBLY WELD

FIGURE 3



END VIEW OF MODULE NO. 7 PRIOR TO FINAL
ASSEMBLY WELD

991C

FIGURE 4

BRAYTON CYCLE TEST MODULE

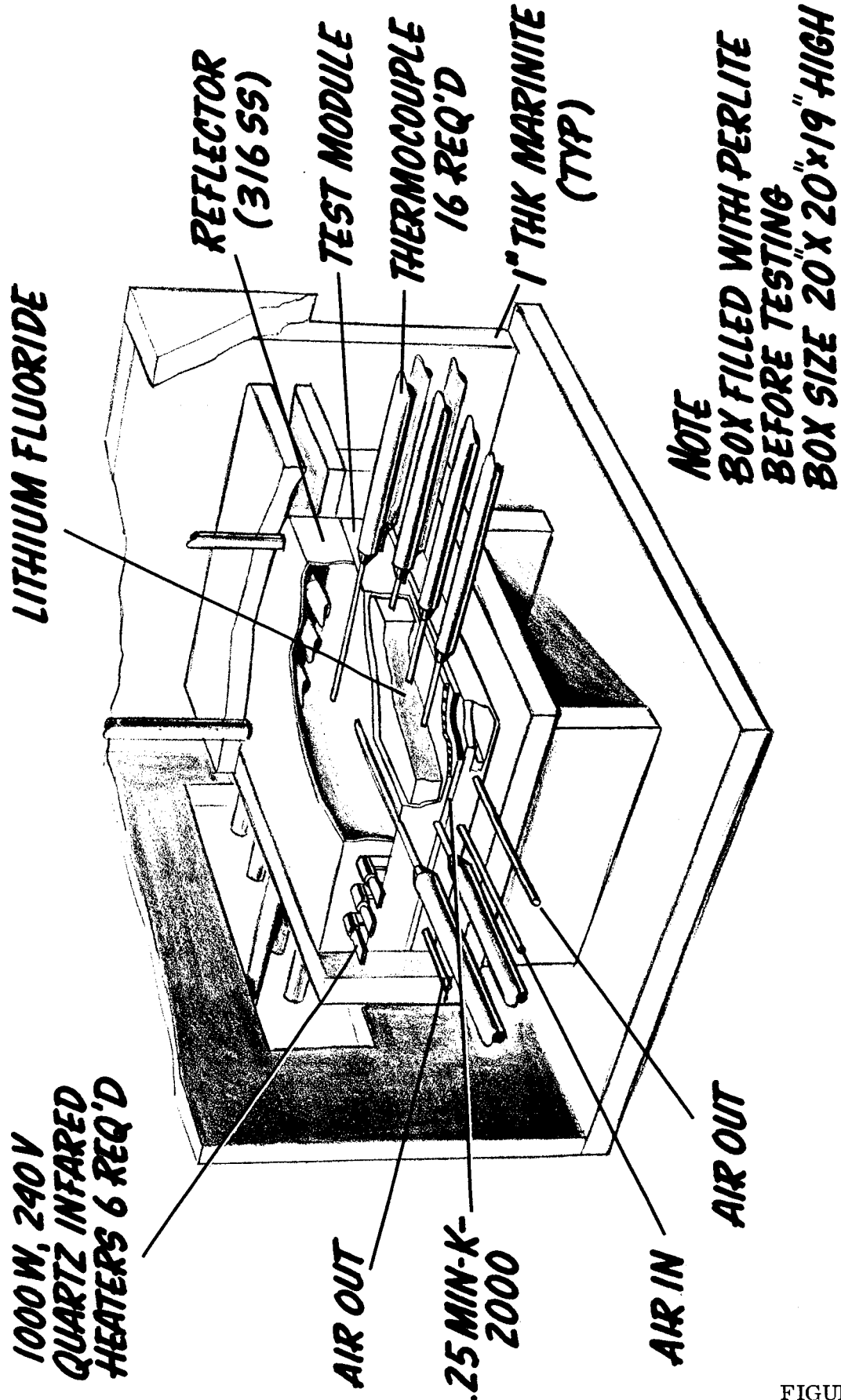
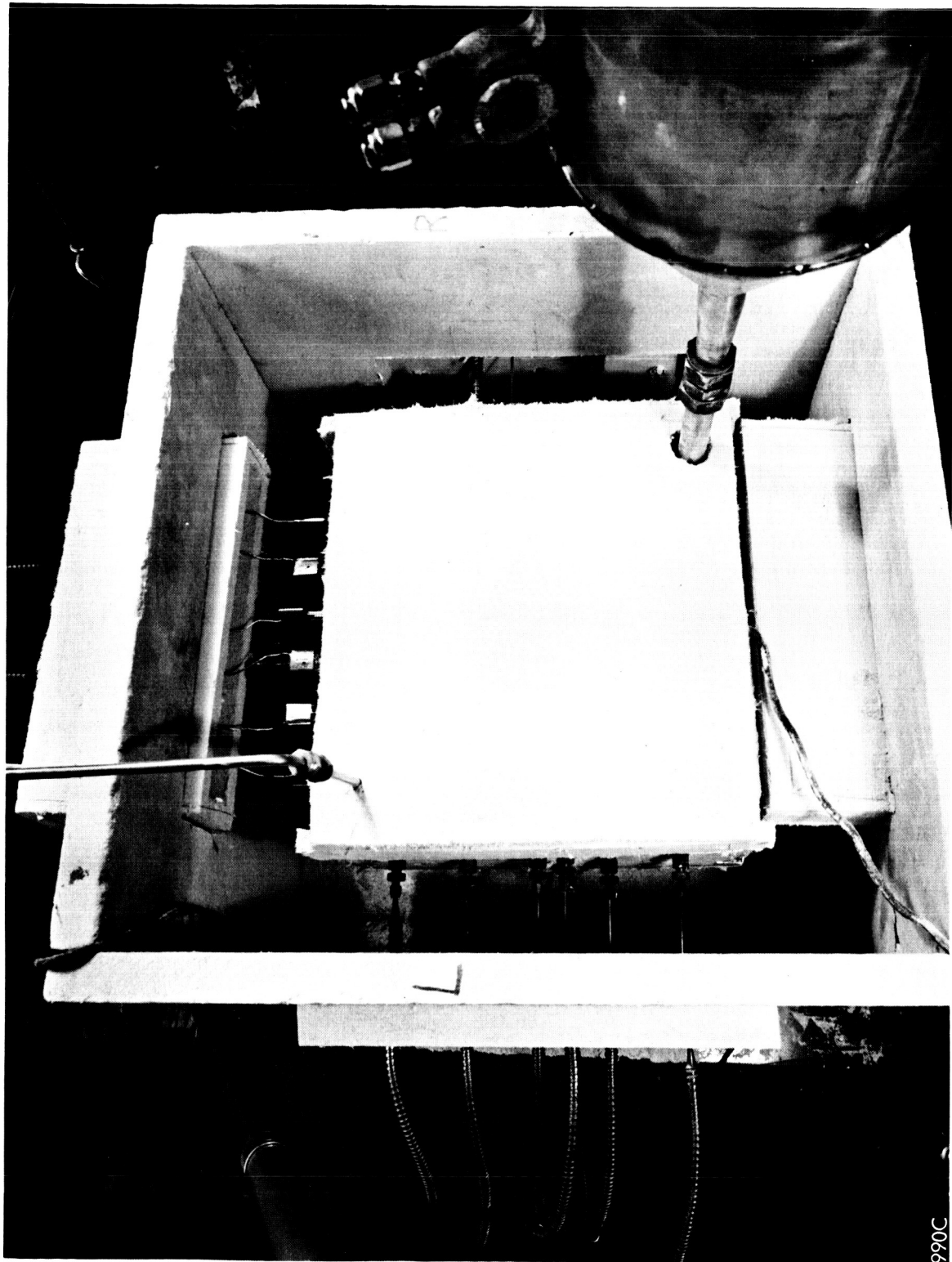


FIGURE 5



TOP VIEW OF TEST BED WITH HEATER ASSEMBLY
IN PLACE PRIOR TO FILLING WITH PERLITE

990C

FIGURE 6

TYPICAL TEMPERATURES MEASURED IN LIQUID THERMAL CONDUCTIVITY TESTS

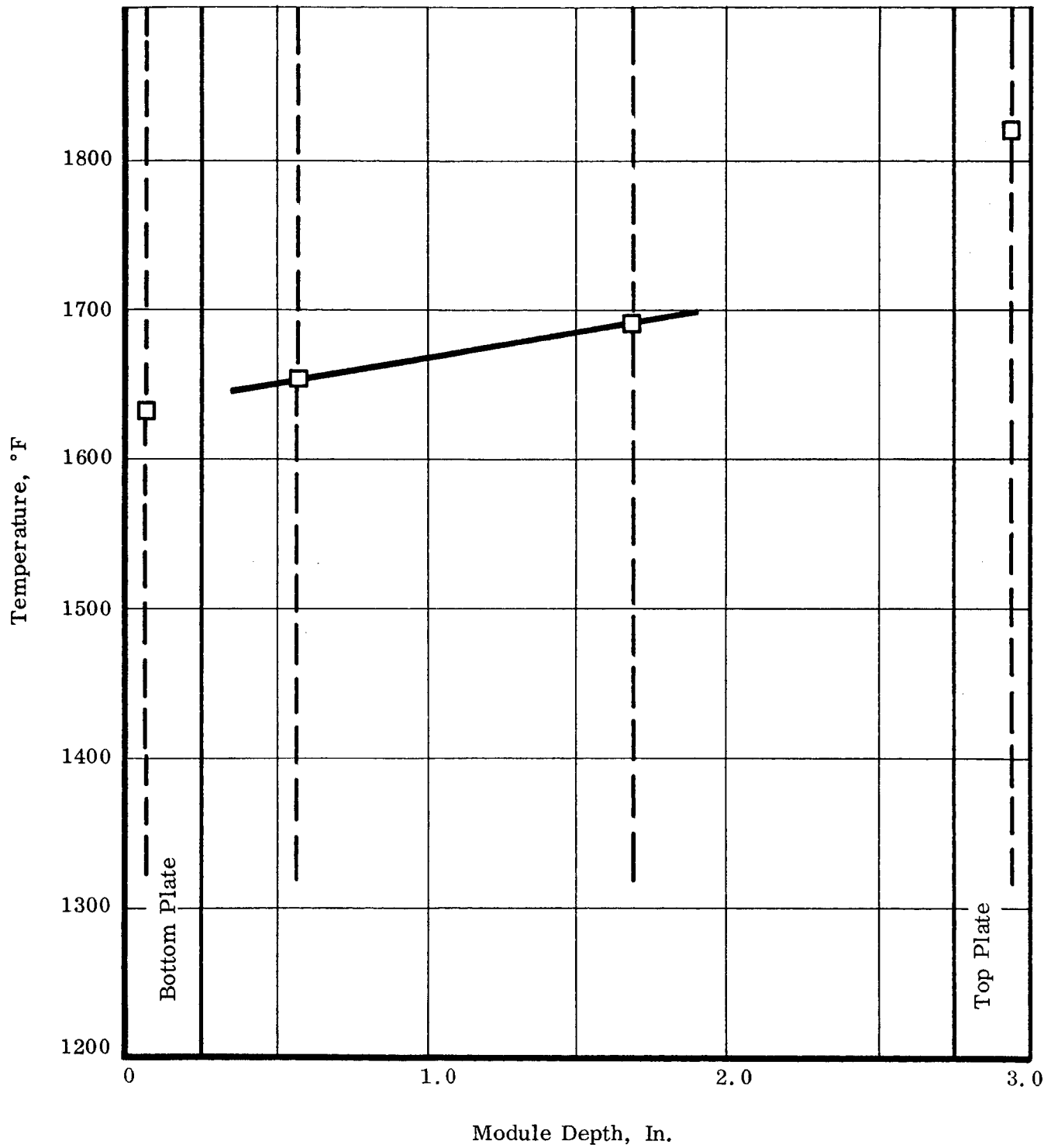


FIGURE 7

TYPICAL TEMPERATURES MEASURED IN SOLID THERMAL CONDUCTIVITY TESTS

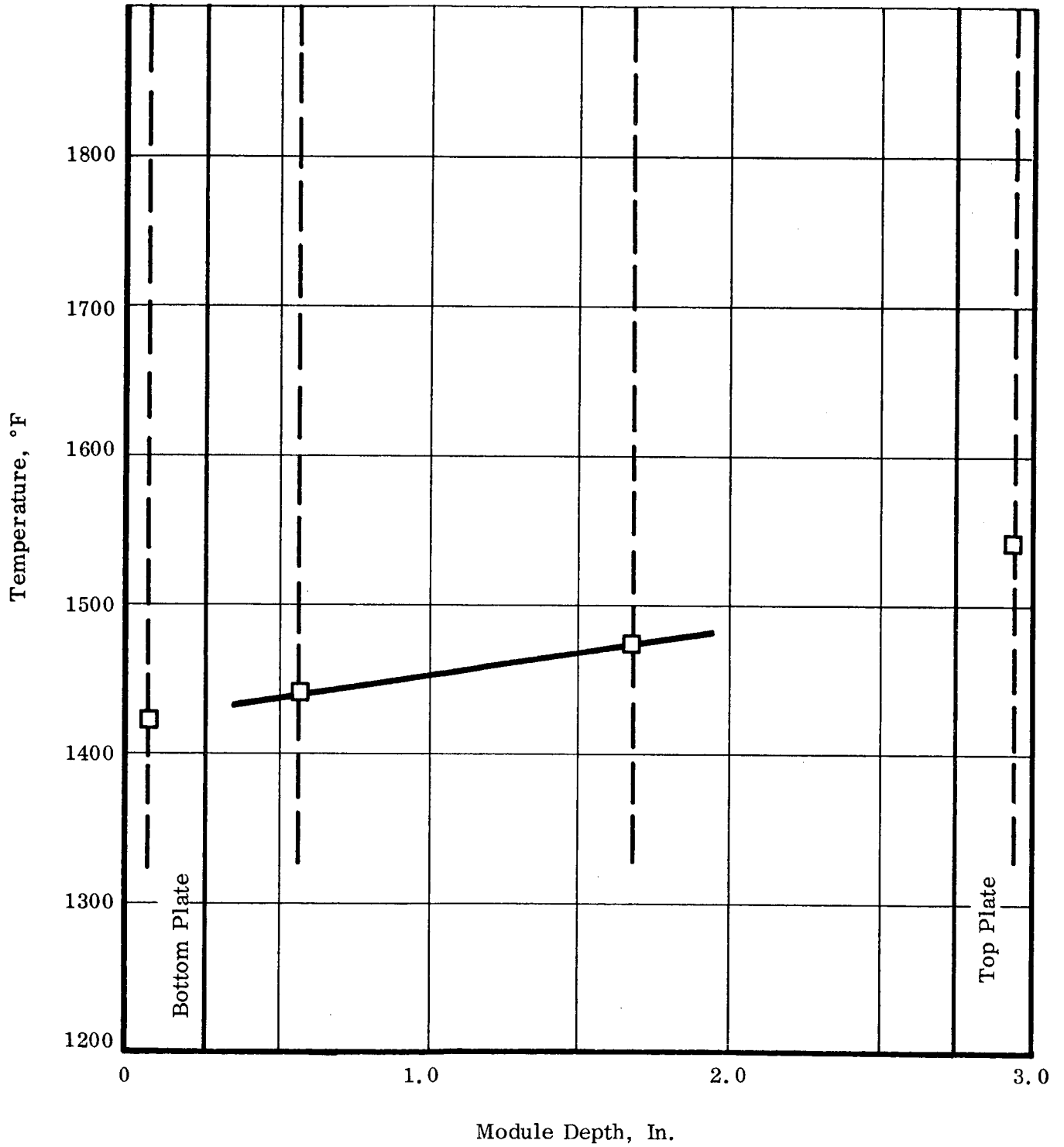


FIGURE 8

In the heating portion of the cycle, the heater power setting was maintained constant at each of three levels. A minimum of three cycles was employed to establish repetitive data. Thus, a minimum of three data points was obtained on each of the modules.

The top plate temperature variation with heating time at several power levels is presented in Figure 9 for module number 5. It can be observed that the time required to melt all of the lithium fluoride decreases with increasing heater power level. The top plate temperature is of interest since it represents the cavity surface temperature in the flight design. In the flight design, the cycle imposed is 60 minutes of sun time and a maximum of 38 minutes of shade time for the design of prime interest to NASA-Lewis. In the above 300 nautical mile orbit, all of the lithium fluoride must be melted at the end of the 60 minute heating time. Therefore, the all melted top plate temperature is representative of the maximum cavity surface temperature anticipated in the flight design at any given heat input flux level.

Preliminary results of all the test data were presented to NASA in the technical meeting of March 5 and 6. Subsequent discussion of these results produced several questions of concern in the data. In particular, the performance of module number 2 was much better than had been anticipated, and in fact showed a lower top plate temperature for a given heat flux level than some of the modules with extended surface. Physically, this situation is very unlikely. Examination of the data for module number 2 was continued, and physical measurements of the sectioned module were taken. A view of the sectioned module is shown in Figure 10. It can be observed from Figure 10 that the top and bottom plates are neither flat nor parallel. In fact, the distance between the plates at the center is 0.3 in. less than the distance at the sides.

Subsequent examination of the top plate temperature on heating revealed that a dip in temperature occurred after about 25 minutes of heating, and the slope of the temperature-time curve was considerably less after the dip than before. A calculation of the height of lithium fluoride at the end of 25 minutes heating time indicated that the lithium fluoride could indeed contact the wall. Under these circumstances, the subsequent heat transfer from the top plate to the storage bath could be by conduction rather than the intended radiation across a definite gap. Further, the bath temperatures at the end of the heating times were generally higher with this module than with the others. Thus, it appears that the low temperature levels reported to NASA were caused by conduction into the liquid lithium fluoride. Because of this situation, the data for module number 2 are not applicable to the case where heat transfer is by radiation only. Hence, it is felt by both NASA and TRW that it is necessary to test another module which will in fact provide heat transfer by radiation only.

The cause of the difficulty with module number 2 was mentioned in the second quarterly progress report, ER-5736. A premature failure of a small bleed tube attached to the top plate permitted liquid lithium fluoride to escape. The fluoride attacked the top, sides and bottom of the module, the quartz heater elements and the Min-K insulation. Before refilling this module with lithium fluoride, it was necessary to machine new grooves in the top plate to house new thermocouple wells. This machining had to be performed on the assembled module. Obviously, the top plate was supported only at

TYPICAL VARIATION OF TOP PLATE TEMPERATURE WITH HEATING TIME
AT SEVERAL POWER LEVELS - MODULE NO. 5

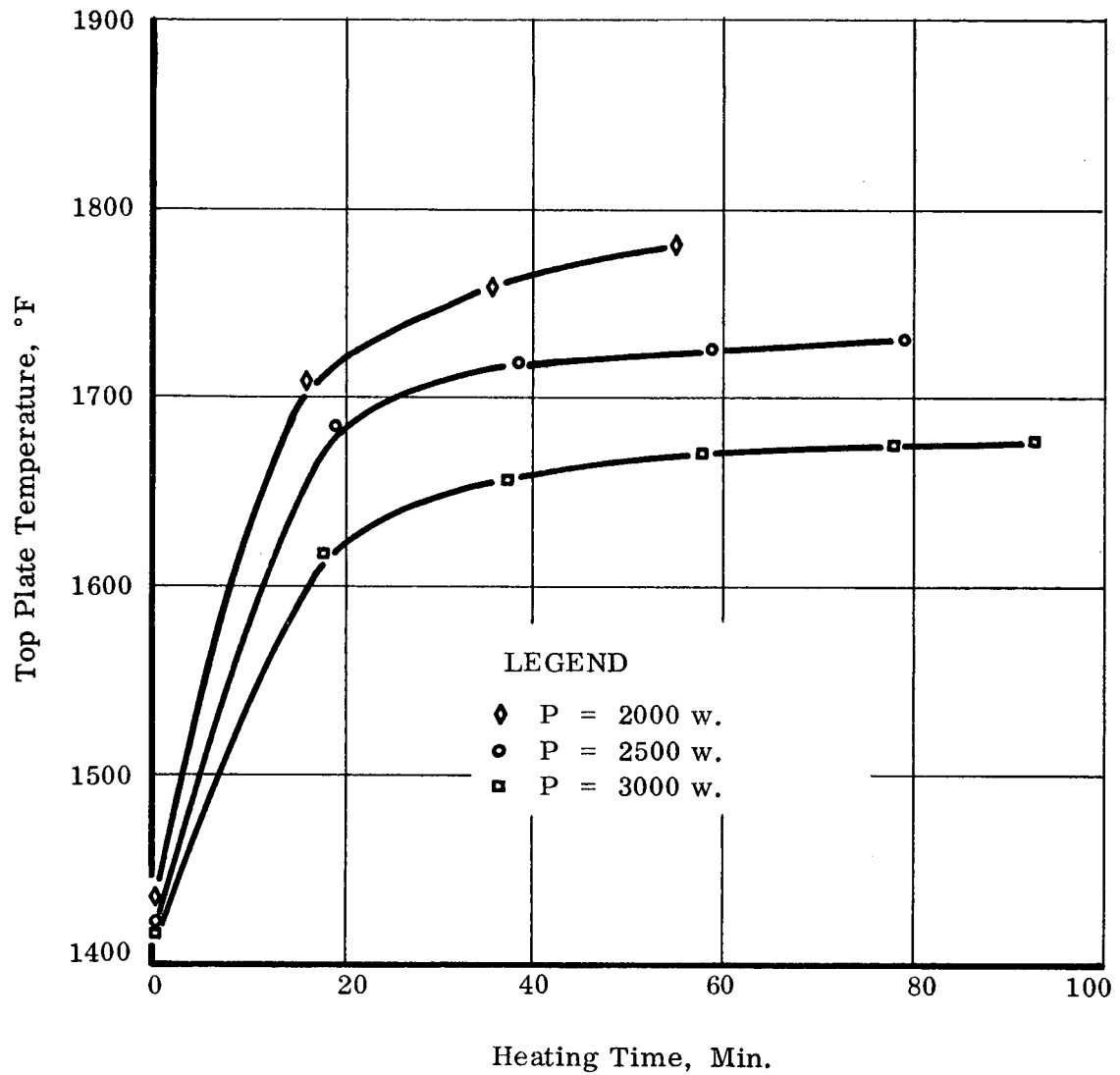
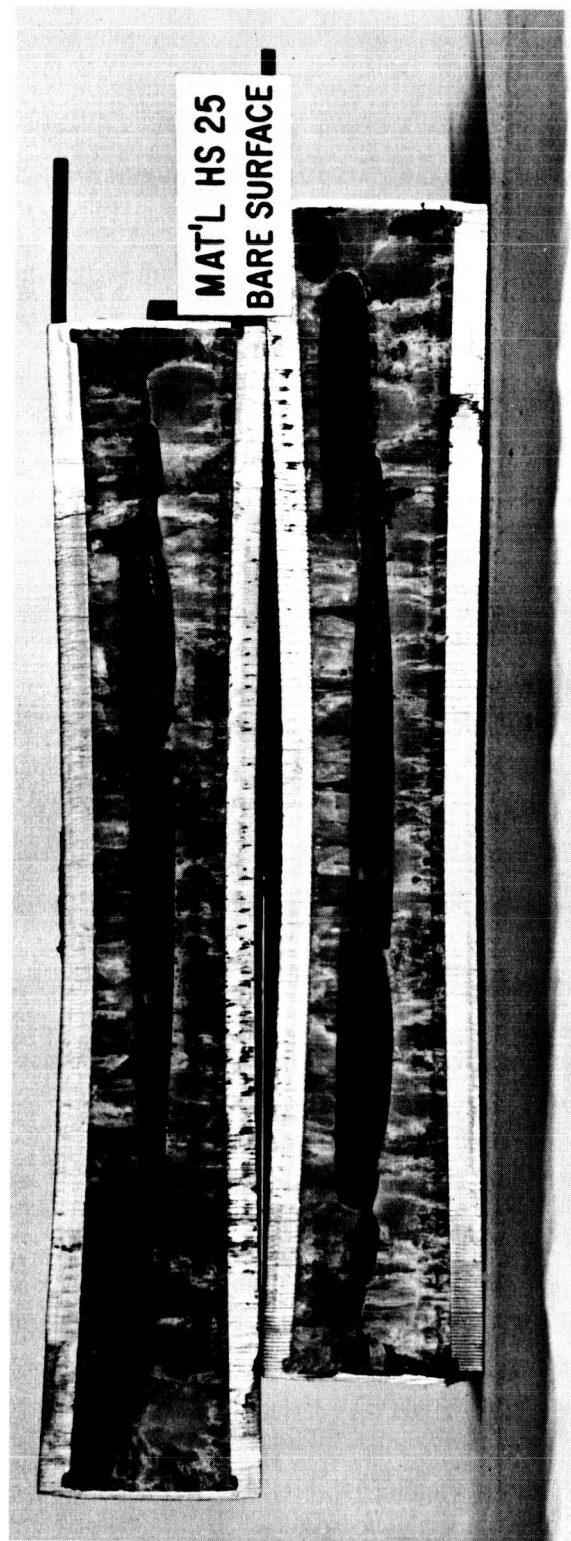


FIGURE 9



VIEW OF MODULE NO. 2 SECTIONED AFTER COMPLETION
OF TESTING

FIGURE 10

edges and was essentially unsupported in the center. After the machining was completed, the test engineer noted that the top surface was no longer flat. It was decided at the time to refill with fluoride and attempt to obtain the proper data. Subsequent events have shown that it was impossible to get the correct data with the modified module.

In the normal assembly of the modules, the top plate is clamped to a press and fully supported during the cutting of the grooves. Thus, no other problems of this nature were encountered in the test program.

A sectioned view of module number 3 is presented in Figure 11. This view is of interest because it shows the characteristic freezing pattern of a heat storage salt around a circular tube. The four tubes with the large spacing were cooled during the last freezing process. The results are obvious.

The top plate in Figure 11 can be observed to be flat. The HS 25 modules, except for module number 2 noted above, held up very well. The sectioned views of the 316 stainless steel modules indicated that the strength of the top plate was marginal at the test temperatures encountered. Thus, all future modules will be constructed of HS 25 alloy, or a similar high strength alloy.

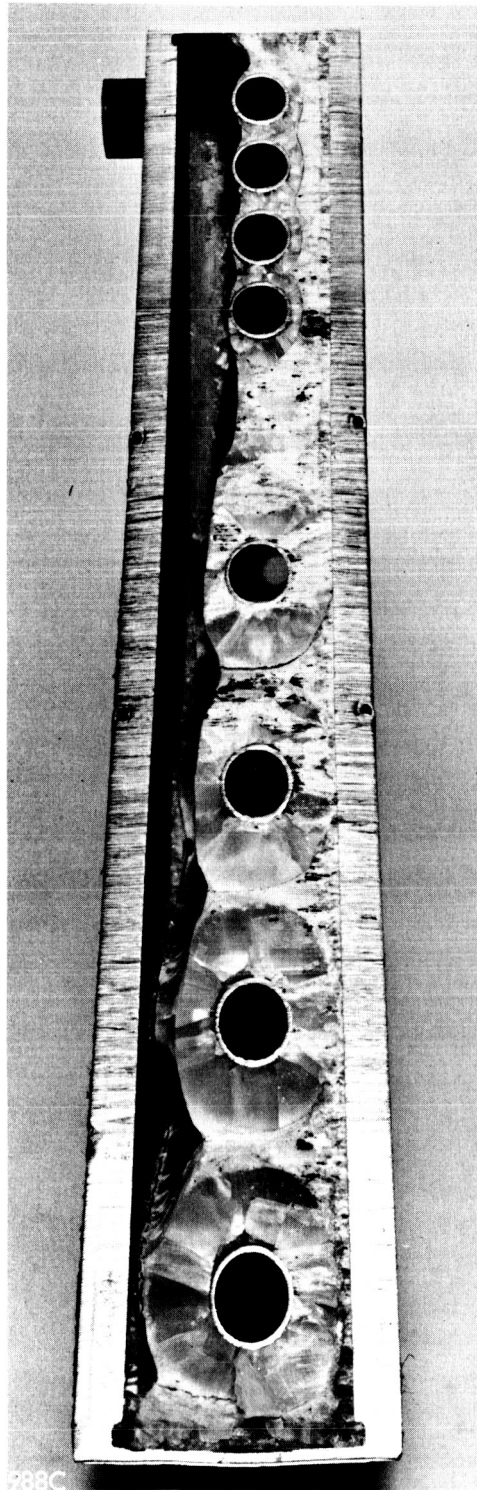
The variation of the top plate temperatures at the end of melting with the heat input flux was presented to NASA, but review of the results within TRW has raised additional questions on the calculation of the heat input flux. These questions must be solved and the answers substantiated before the results can be accepted as valid. In view of these questions, it is preferred not to publish the results until a sound basis for the calculation of the heat input flux has been firmly established.

Metallurgical examination of the failed bleed tube from module number 2 was unable to identify the primary cause of failure. Results did show that the tube O. D. was much more severely attacked than the tube I. D. The wall thickness was reduced from 0.029-in. to between 0.005 in. to 0.010 in. The external attack was in the form of grain boundary penetration, followed by complete destruction of the grains. From visual inspection, the outside surface was extremely scaled. X-ray diffraction analysis of the scale indicated that the composition of the scale was a combination of fluorides and spinel oxides.

The attack on the inside of the tube was apparently caused by the exposure to molten lithium fluoride. The inside of the tube underwent grain boundary penetration to a maximum depth of 0.005 in. As contrasted to the tube outside, little or no scaling occurred on the inside. The internal attack was similar to that encountered in failures in Task II discussed later in this report.

3.1.4 Flight Type Design

The first step in arriving at a flight type design from the results of the preliminary design analysis was the review and evaluation of the three potential concepts evolved



VIEW OF MODULE NO. 3 SECTIONED AFTER COMPLETION
OF TESTING

FIGURE 11

in the preliminary design concepts. These concepts were presented in the second quarterly progress report, ER-5736, and will not be repeated here. Engineering, design, manufacturing, reliability, quality assurance and materials personnel contributed to various portions of the design review and subsequent evaluation. The entire process resulted in the selection of an optimum shape for use in the flight type design. To aid in the evaluation, numerical ratings were assigned to each of the three shapes in several important areas. A summary of these ratings is included below.

CONTAINER EVALUATION

<u>Item</u>	<u>Hemisphere</u>	<u>Cone</u>	<u>Shape</u> <u>Cylinder</u>
Simplicity	3	2	1
Fabrication	3	2	1
Performance			
Thermal	1	2	3
Stress	1	2	3
Cavity	1	2	3
Overall			
Reliability	1	2	3

The numerical ratings are not truly indicative of the relative position of each shape in that the difference between a number 1 and a number 2 could be very slight and the difference between the number 2 and the number 3 could be quite large. Thus, a completely true picture cannot be obtained from the above.

In the cavity performance area, for example, preliminary calculations indicated that the cylinder could not retain sufficient energy to meet the heater duty required, and the cone was marginal in this respect. Since this type of calculation is known to give optimistic results compared to the more detailed calculations, both the cylinder and the cone were essentially eliminated on the basis of poor or marginal cavity performance. A popular analytical variation of the cone, that of an inverted cone with an internal conical section for multiple reflections, was not seriously considered for this application because of the complexity involved and the ultimate requirement for an all sun orbit.

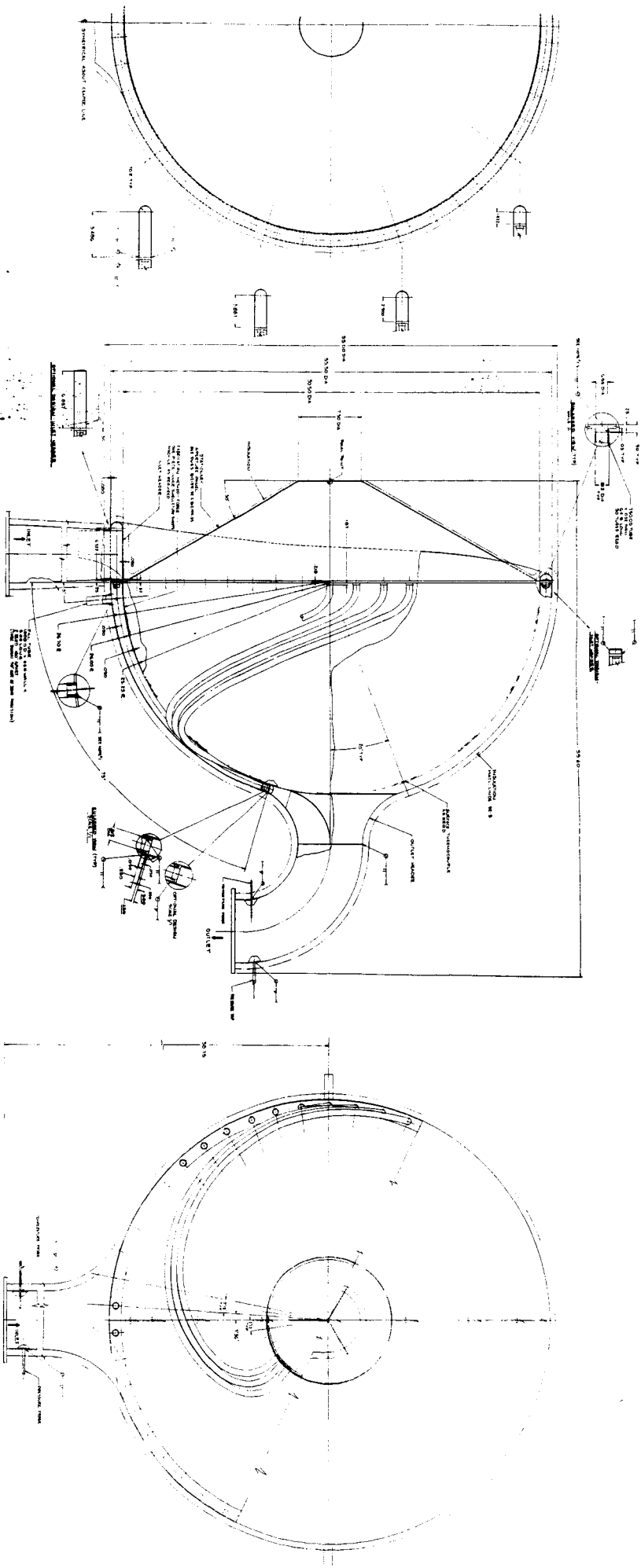
One of the most serious objections to the hemispherical design resulting from the preliminary design studies was the large number of tubes (130) specified. Reliability, manufacturing and management personnel were very unhappy with such a large number. After the hemisphere had been selected as the optimum shape, a further design effort was made to reduce the number of tubes required to as low a value as practical. The limit is set by the heater tube characteristics as determined from NASA specifications and the free lithium fluoride volume required.

The new design effort resulted in a design with the following characteristics:

Tube Diameter:	0.684 in.
Tube Length:	5.8 ft.
Number of Tubes:	50
Cavity Diameter:	51.0 in.
Maximum Annular Thickness:	1.4 in.
Gas Weight Flow:	36.7 lb/min.
Allowable Gas Pressure Drop:	0.55 psi

The above design was made for the combination of a 30-ft diameter collector and a nominal 300 nautical mile earth orbit. The tentative system specifications for this combination and three others were provided by NASA and are listed in Table I of the second quarterly report, ER-5736. The results for the other combinations are presented later.

The layout of the basic container for the above combination is shown in Figure 12. The cavity surface temperature control and aperture closure device is a necessary and integral part of the cavity receiver component. The concept which was evolved from a study of the requirements is illustrated in Figure 13. The temperature control concept features the use of liquid metal contained within a closed system consisting of a temperature sensor and a bellows actuator. The vapor pressure of the liquid metal increases with temperature, and as the desired maximum temperature is approached, the vapor pressure produces a force sufficient to actuate the bellows. Linkages attached to the bellows open doors and increase the cavity reradiation losses, thus reducing the cavity surface temperature. A similar type of system is proposed for the aperture closure device. The results of the study and the concept in Figure 13 were presented to NASA on March 5 and 6. NASA technical personnel felt that the design shown was too complex and cumbersome. They suggested added effort should be performed to arrive at a design suitable for hardware development. A good start was made in this direction during March and early April, but the remaining contract funds are low and this effort must be stopped until additional funding is available.



CAVITY RECEIVER 300 NM - 30 FOOT MIRROR
BRAYTON CYCLE

In conjunction with the flight type design effort, a manufacturing process study was conducted to investigate the potential fabrication processes, and sources available for the full scale unit. Of the various parts needed to be fabricated for the basic container, the tubes, inlet header, outlet header, inlet transition and outlet transition are state-of-the-art applications and present few problems. The inner and outer shells present considerable difficulties, particularly if fabricated in one piece. The problem on these parts is twofold:

1. Material Availability
2. Method and Source Selection

The high temperature non-refractory materials considered for this application are:

Haynes 25

Haynes 56

Hastelloy X

TD Nickel

Considerable effort has been expended to locate sources of ingot and rolling mills willing to handle a small order. Two sources were finally found. One source was willing to roll these materials, except TD nickel, up to 72 in. square. The other source quoted a firm price for 3/8 in. plate to 65 in. square. The exception for TD nickel is caused by the fact that it is a proprietary material not presently available in sheet above 24 in. width. Discussions are in progress with DuPont to develop a program plan necessary to provide larger width sheet.

Three processes have been considered for these shells:

1. Forging
2. Shear Spinning
3. Hand Spinning

As of press time, no vendors have been willing to quote on a forging because of lack of material availability in the proper size and plate thickness. One vendor has submitted a program plan for development of shear spinning. Two vendors have quoted on hand spinning. Out of a list of over 45 vendors contacted, all others have declined to quote for various reasons.

A stress analysis effort was conducted as part of the flight type design. This effort had been geared to provide as much stress information as possible in the topical report due 1 April 1964. At the time of the NASA direction stopping all work on the topical report, this effort was discontinued. The stress investigations conducted up to that time had indicated that considerable effort is needed to select and substantiate the proper stress model for each of five joints in the flight type design. It is anticipated that this effort will be performed as part of the additional work requested by NASA.

Although the major emphasis was placed on the above design, similar concepts were calculated for each of the other three combinations of interest to NASA. Also, off-design performance was investigated and designs for a range of allowable gas pressure drops were estimated. These items are covered in the next section.

A reliability design review was conducted as part of the prime design effort above. The reliability portion is discussed in Section 3.1.6.

3.1.5 Alternate Design Studies

The essential specifications for the other three combinations are taken from Table I of ER-5736 and listed below:

<u>Item</u>	<u>Conditions</u>		
	<u>1</u>	<u>2</u>	<u>3</u>
Mirror Diameter, ft.	20	20	30
Orbit	300 N. M.	Sync.	Sync.
Heater Inlet Pressure, psia	6.10	9.24	20.79
Heater Outlet Pressure, psia	5.85	8.86	19.95
Mass Flow, lb/min	16.31	24.72	55.62
Power Input, kw	18.24	27.65	62.21
Net Power Output, kwe	3.56	5.39	12.13

The design studies for these three conditions resulted in hemispherical concepts similar to Figure 12 but with the following characteristics:

<u>Item</u>	<u>Conditions</u>		
	<u>1</u>	<u>2</u>	<u>3</u>
Number of Tubes	70	30	30
Tube Diameter, in.	0.597	0.910	0.910
Tube Length, ft.	4.18	7.53	8.64
Cavity Diameter, ft.	3.75	4.12	5.17
Estimated Weight, lb.	236	440	830

It can be observed that the practical number of tubes is reduced with the increased free lithium fluoride volume requirements of the synchronous orbit.

Typical results for the off-design performance calculations are illustrated in Figures 14 and 15. Figure 14 shows the gas temperature variation encountered with an instantaneous change of ten (10) percent in the gas flow rate. Figure 15 shows the gas pressure variation under the same conditions.

The design investigation of perhaps the most interest to NASA systems personnel was an examination of the effect a change in allowable gas pressure drop might have on the cavity receiver design. It can be observed from the list of conditions above that the allowable gas pressure drop in the heater had been set at approximately four (4) percent of the heater inlet pressure. NASA requested that designs based on allowable gas pressure drops of two, four and six percent of the heater inlet pressure be studied. The results are shown in Figure 16 for the combination of the 30-foot diameter collector and the 300 nautical mile orbit. It can be observed from Figure 16 that the estimated weight for the two percent design is about 30 pounds more than the four percent design, but no further weight further reduction is achieved at six percent. One requirement for the two percent design is a larger cavity diameter and thus a larger amount of collector blockage. Despite this disadvantage, NASA is anxious to pursue the two percent design to a full layout. If no additional disadvantages are uncovered, the two percent design should be very attractive to the systems designers in that the lowered pressure drop permits a tolerance for off-design performance on the part of the turbomachinery.

3.1.6 Reliability

The reliability program specified by the contract work statement was completed. The reliability plan was transmitted to NASA. A reliability design review was conducted and many questions were posed on the mechanical complexity of the aperture closure and cavity surface temperature control device. These questions were of the same general type later raised by NASA personnel. Finally, the reliability personnel made a prediction of the failure rates and subsequent reliability.

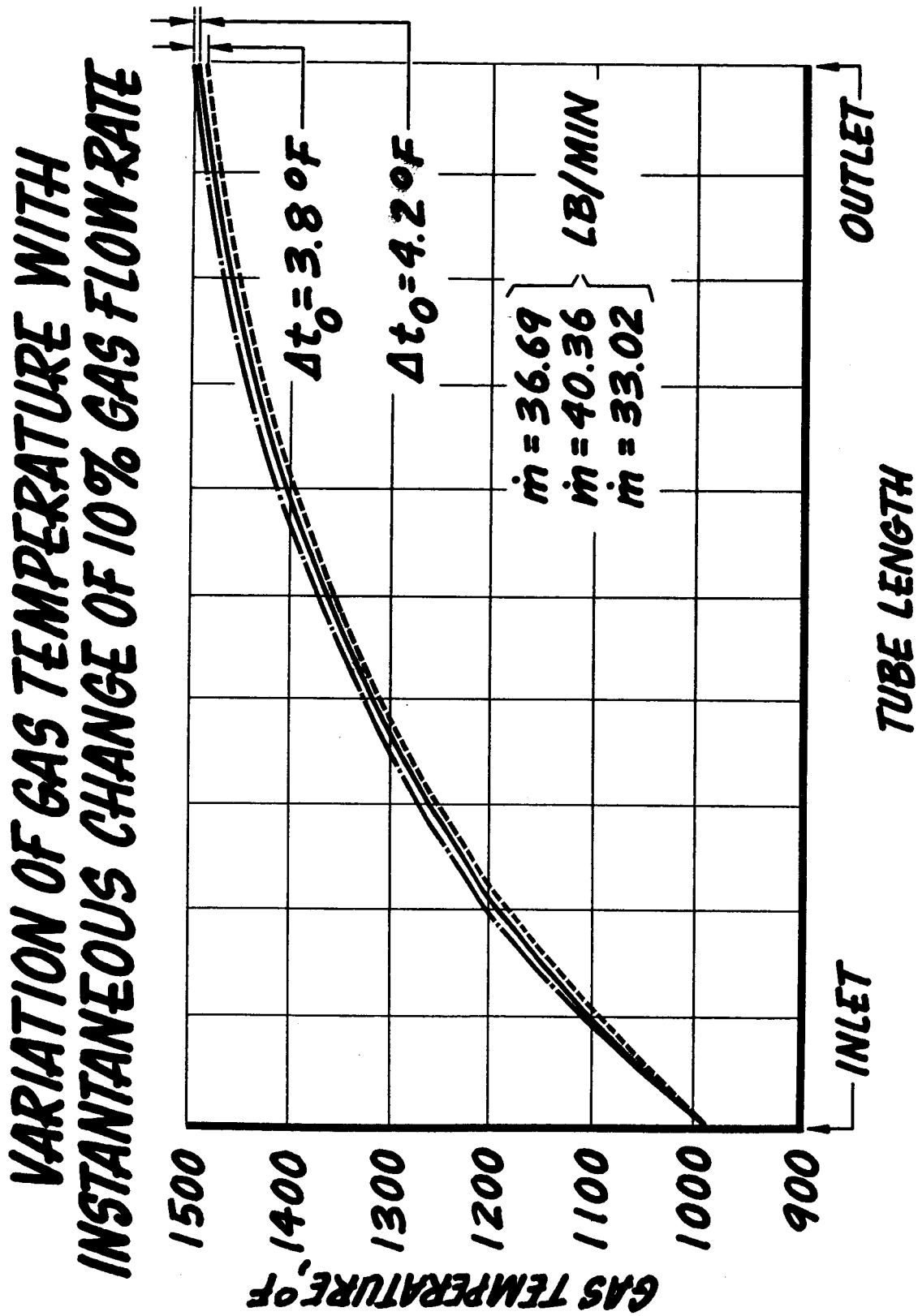


FIGURE 14

VARIATION OF GAS PRESSURE DROP WITH INSTANTANEOUS CHANGE OF 10% IN GAS FLOW RATE

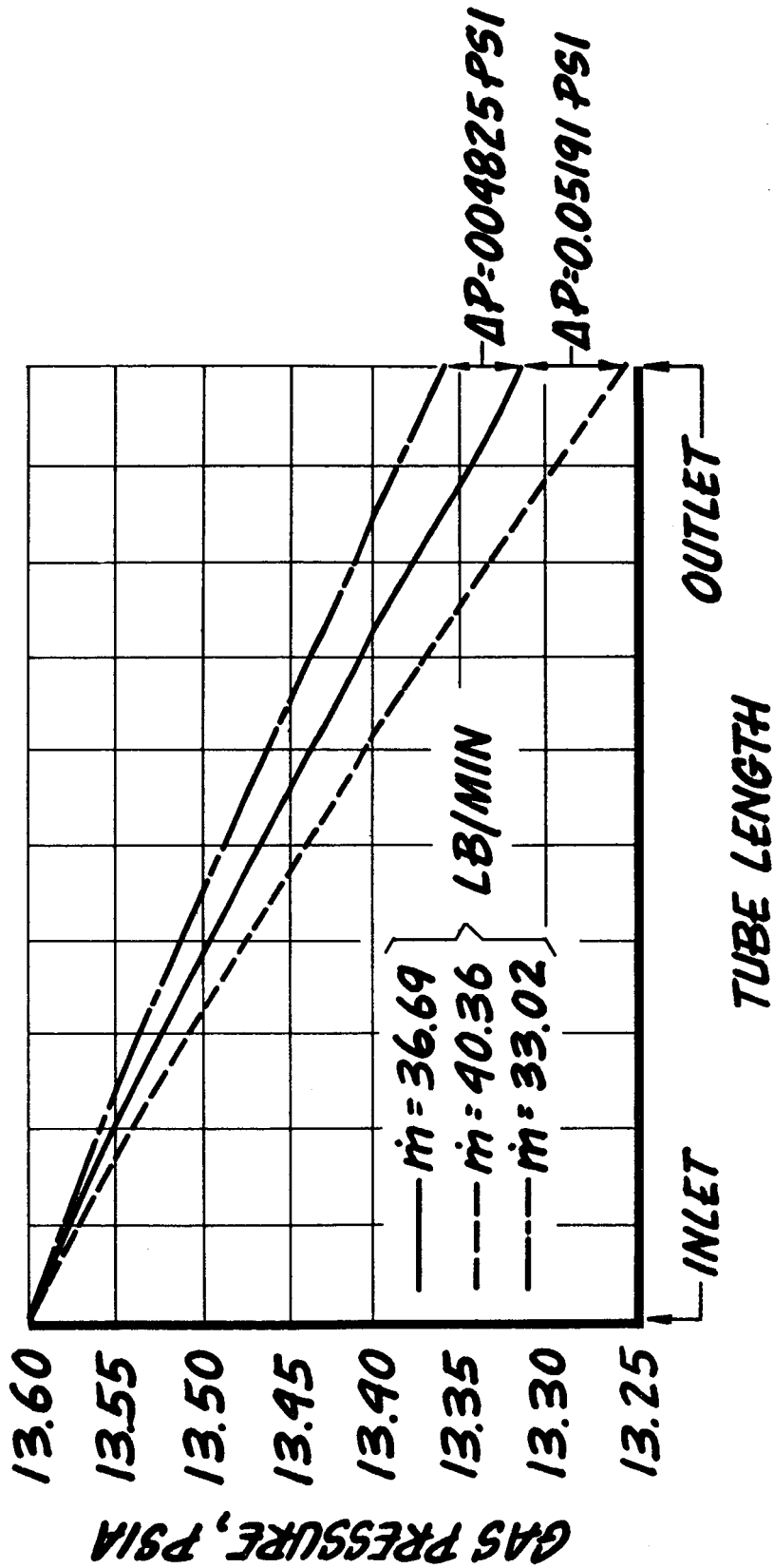


FIGURE 15

EFFECT OF $\Delta P/P_{IN}$ ON RECEIVER WEIGHT

300 N.M. ORBIT, 30 FOOT MIRROR

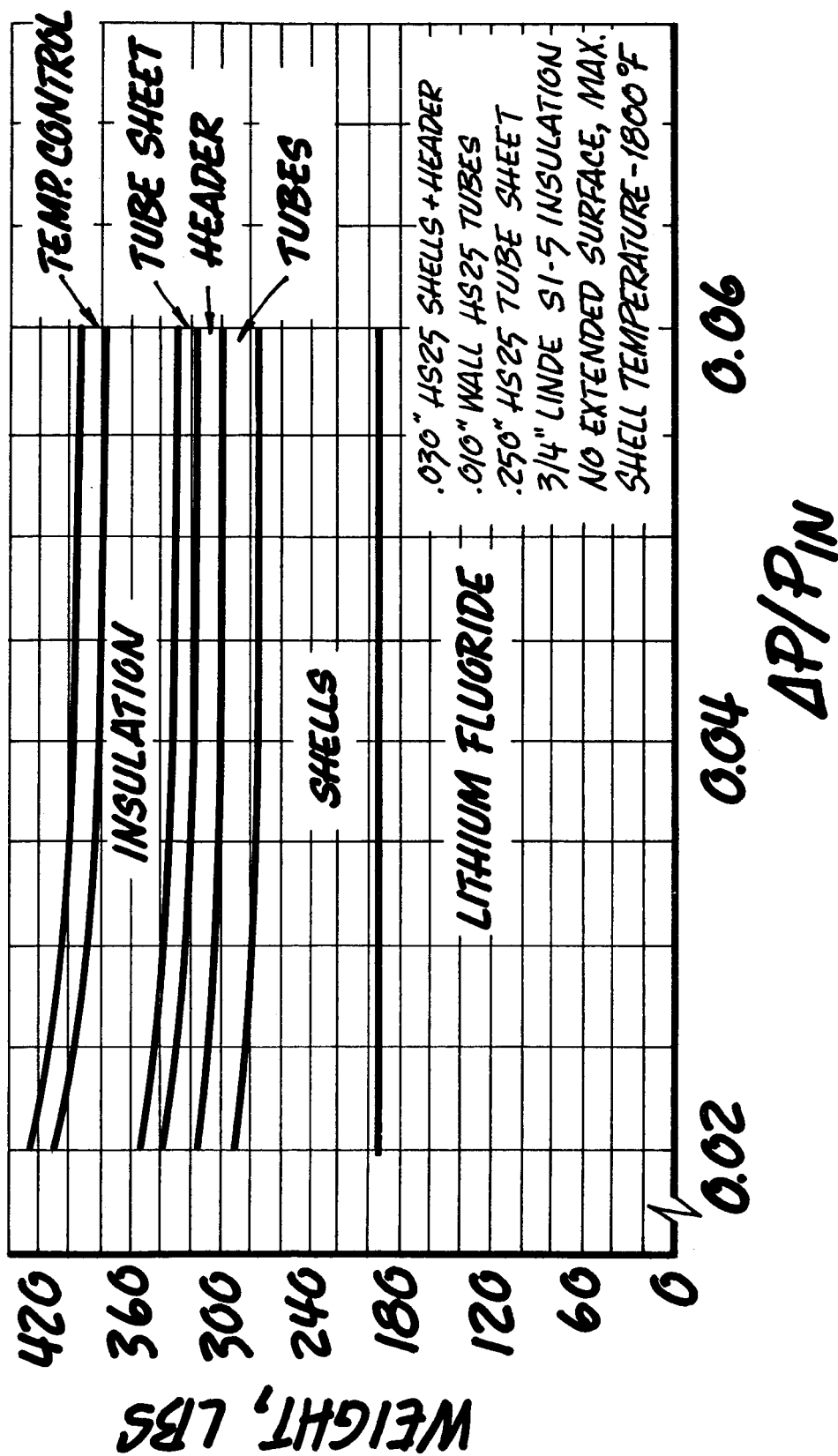


FIGURE 16

3.2 Task II Materials Compatibility Investigation

As discussed in the previous quarterly progress report (ER-5736, Brayton Cycle Cavity Receiver, Quarterly Report for period ending December 1963), the first 2500 hour capsule test was interrupted after 672 hours because of capsule failures. Since that first time, the test has been interrupted two more times because of capsule failures. The second interruption occurred after 1372 hours and the third after 2175 hours. In each case, the capsule container, fabricated from 1/8 inch Hastelloy X, suffered attack from the lithium fluoride escaping from the capsules. Figure 17 shows the extent of damage incurred by the capsule container which failed after 672 hours. Indication of a capsule failure is the loss of argon flow through the exit tube of the capsule container. This loss of flow occurs when the container walls are penetrated by the lithium fluoride, thus allowing the argon to escape freely through the can.

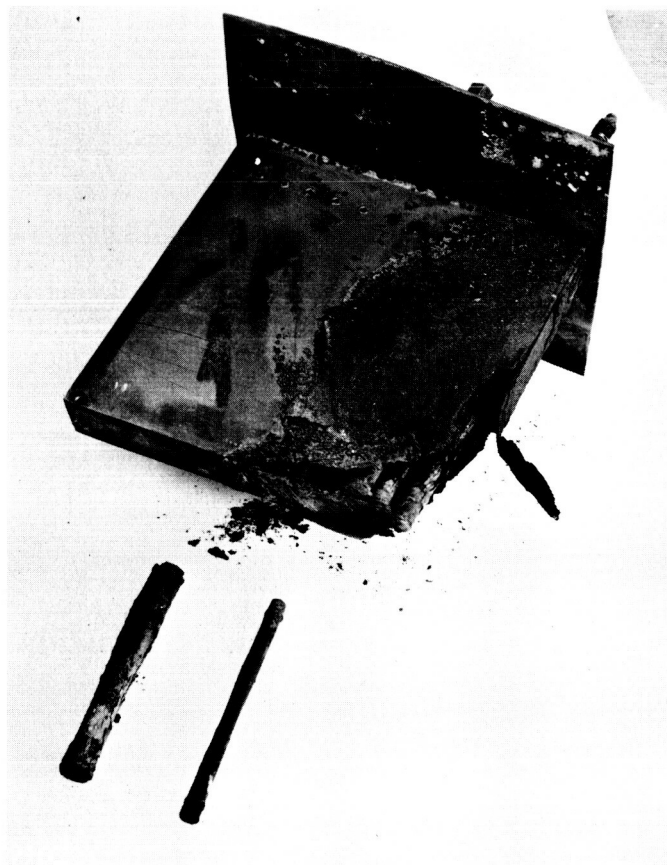
A summary of the interruption and action taken after each is given below:

SUMMARY OF INTERRUPTIONS

<u>No.</u>	<u>Hours</u>	<u>Capsules Removed</u>	<u>Capsules Added</u>
1	672	4 Rene 41	2 Waspaloy, 2 Hastelloy N
2	1372	1 Rene 41, 2 Udimet 500, 1 TD Nickel	None
3	2175	2 TD Nickel	None

As of report time, the test is once again in progress. It is intended to complete the remaining 325 hours of the desired 2500 hours of test time before removing any capsules which are believed to be sound. The present status of the capsules under test or removed from test is shown in Table I.

Identification of the capsules which failed after 672 hours of operation was made by visual inspection. As shown in Figure 18, all three capsules were severely corroded. These capsules (No's 6, 7 and 8) were fabricated from 3/4 inch I. D. by 0.62 inch wall tubing with longitudinal seam welds. In view of the severity of attack incurred by the three capsules, a fourth Rene 41 capsule (No. 5), which did not fail, was also extracted for examination. This capsule was likewise fabricated from the 3/4 inch I. D. tubing. However, it did not contain the longitudinal seal weld. All four of the capsules were welded in the solution treated condition. Rene 41 was used as the filler material. The capsules were tested in the as-welded condition.



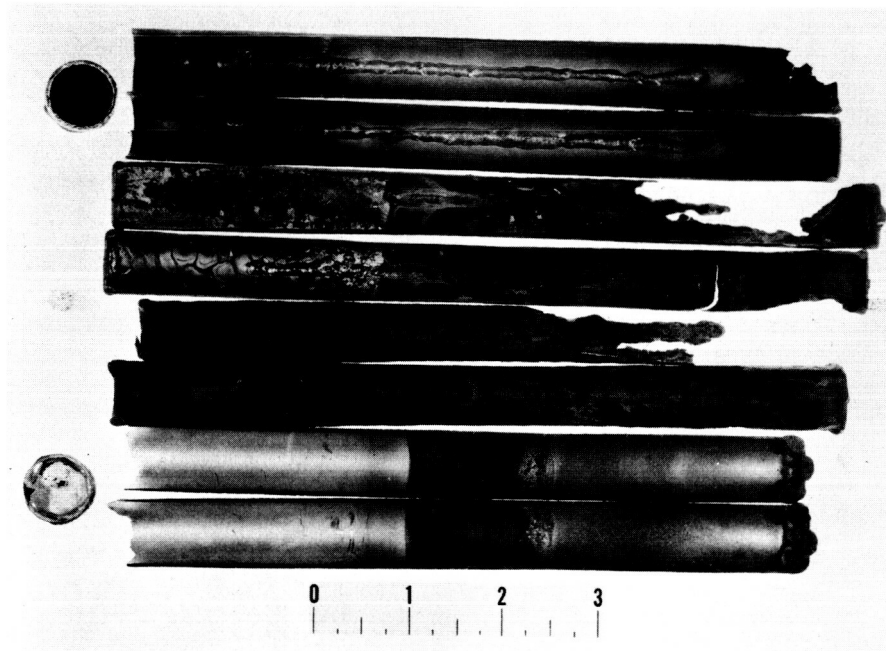
07554-4

APPEARANCE OF CAPSULE CONTAINER WHICH FAILED AFTER
672 HOURS

FIGURE 17

TABLE I
STATUS OF CAPSULES PLACED INTO FIRST 2500 HOUR TEST

Capsule No.	Material	Welded End Caps	Longitudinal Seam Welds	Brazed End Caps	Welding Filler Material or Braze Material	Time Accumulated as of Midnight April 13 (hrs)
2	Rene 41	X			Rene 41	Removed after 1327 hrs.
5	Rene 41	X			Rene 41	Removed after 672 hrs.
6	Rene 41	X	X		Rene 41	Failed after 672 hours
7	Rene 41	X	X		Rene 41	same
8	Rene 41	X	X		Rene 41	same
10	Udimet 500	X	X		Rene 41	Failed after 1327 hours
11	Udimet 500	X	X		Rene 41	same
14	Haynes 25	X			Haynes 25	In test
15	Haynes 25	X	X		Haynes 25	In test
16	Haynes 25	X	X		Haynes 25	In test
17	TD Nickel	X	X		Rene 41	Failed after 2175 hours
18	TD Nickel	X	X		Rene 41	Failed after 1327 hours
19	TD Nickel	X			Rene 41	In test
20	TD Nickel				GE-J8400	Failed after 2175 hours
21	TD Nickel			X	GE-J8400	In test
22	Hastelloy X	X	X		Hastelloy X	2325
23	Hastelloy X	X	X		Hastelloy X	2325
24	Hastelloy X	X			Hastelloy X	2325
25	Haynes 56	X	X		Haynes 56	2325
26	Haynes 56	X	X		Haynes 56	2325
27	Waspaloy	X	X		Waspaloy	2325
28	Waspaloy	X	X		Waspaloy	Placed into test after 672 hours
29	Hastelloy N	X	X		Hastelloy W	Placed into test after 672 hours
30	Hastelloy N	X	X		Hastelloy W	Placed into test after 672 hours



Capsule No. 8

Capsule No. 7

Capsule No. 6

Capsule No. 5

07599

APPEARANCE OF RENE 41 CAPSULES REMOVED FROM TEST
AFTER 672 HOURS OF OPERATION

Four new capsules were prepared to replace the Rene 41 capsules removed, to keep twenty capsules under test. Two of these were made from Waspalloy. The other two were from Hastelloy N. All four capsules had welded end caps and longitudinal seam welds. Because of procurement problems, the Hastelloy N capsules were welded with Hastelloy W filler wire. Waspalloy filler was used for the Waspalloy capsules.

The test was restarted and continued until it was again shut down after 1327 hours because of apparent capsule failures. However, this time the capsule failure or failures were not as easily discernible.

Examination of the capsules by both visual and dye-penetrant methods did not reveal any obvious flaws. In view of this, x-radiographs of the capsules were obtained. The radiographs of the capsules were made at 0° and 90° to the longitudinal seam welds. Examination of these radiographs did not reveal any obvious flaws. However, indications were found that four of the capsules were either devoid or low in LiF. The other sixteen looked good.

The four capsules which looked suspicious were removed from the test, and the test was restarted. The capsules which were removed were:

<u>Capsule Number</u>	<u>Material</u>	<u>Type of Capsule</u>
2	Rene 41	Welded end caps.
10	Udimet 500*	Welded end caps and longitudinal seam welds.
11	Udimet 500*	Same as capsule no. 10.
18	T. D. Nickel*	Same as capsule no. 10.

* Filler material used was Rene 41.

After 2175 hours of operation, the capsule test was stopped for the third time as the result of failures. Again the capsule failure or failures were not obvious. However on the basis of radiographic inspection two capsules were removed from test. Both were fabricated from TD Nickel (capsules Nos. 17 and 20). Capsule No. 17 had welded end caps and longitudinal seam welds. The filler material used for welding was Rene '41.

Capsule No. 20 had both end caps brazed in place. The braze material used was G. E. alloy J 8400 (21 w/o Cr, 8 w/o Si, 21 w/o Ni, .80 w/o B, .4 w/o C, 4.0 w/o W, Bal Co.). Both capsules are presently under examination.

3.2.1 Results of Examination After the 672 Hours Interruption

Results of the metallographic examination specimens removed from the four Rene 41 capsules are listed in Table 2. The three capsules which failed were too severely damaged to obtain any meaningful corrosion data. However some general observations

TABLE II

RESULTS OF EXAMINATION OF NICKEL BASE ALLOYS EXPOSED TO LITHIUM FLUORIDE BETWEEN 1500°F AND
AND 1850°F TEST CONDITIONS

Capsule No.	Material	Exposure Time, Hr.	Average Heat Cycle Hr/Cycle	Average Cool Cycle Hr/Cycle	
5	Rene 41	672	1.0	0.515	Intergranular penetration to a maximum depth of 0.005 inch.
6	Rene 41	672	1.0	0.515	Capsule too severely damaged to obtain any meaningful corrosion data.
7	Rene 41	672	1.0	0.515	Same as Capsule No. 6.
8	Rene 41	672	1.0	0.515	Same as Capsule No. 6.
2	Rene 41	1327	0.995	0.498	Grain boundary penetrations to less than 1 mil.
10	Udimet 500	1327	0.995	0.498	Corrosive penetration of the cracks surface found in the tube wall.
11	Udimet 500	1327	0.995	0.498	Same as Capsule No. 10.
18	TD Nickel	1327	0.995	0.498	Weld contained a crack penetrating the bead with .004 inch corrosive attack to the bead. Less than .001 inch attack to the TD Nickel.

can be made. All three capsules suffered attack in varying degrees along the ID surface. The attack was in the form of grain boundary penetration and leaching. The depth of attack varied from less than 0.001 inch up to a maximum of .010 to .012 inch. Figure 19, a photomicrograph of a specimen removed from the bottom of capsule 8, illustrates the appearance and depth of attack incurred. In all cases, the attack incurred by the outer surface of the tube was heavier than that suffered by the inside surface. Also with respect to severity of attack the weld beads and the weld affected areas appeared about the same as the rest of the capsule wall. Thus it does not appear that the welds or weld affected areas were any more susceptible to attack than the rest of the Rene 41 capsules.

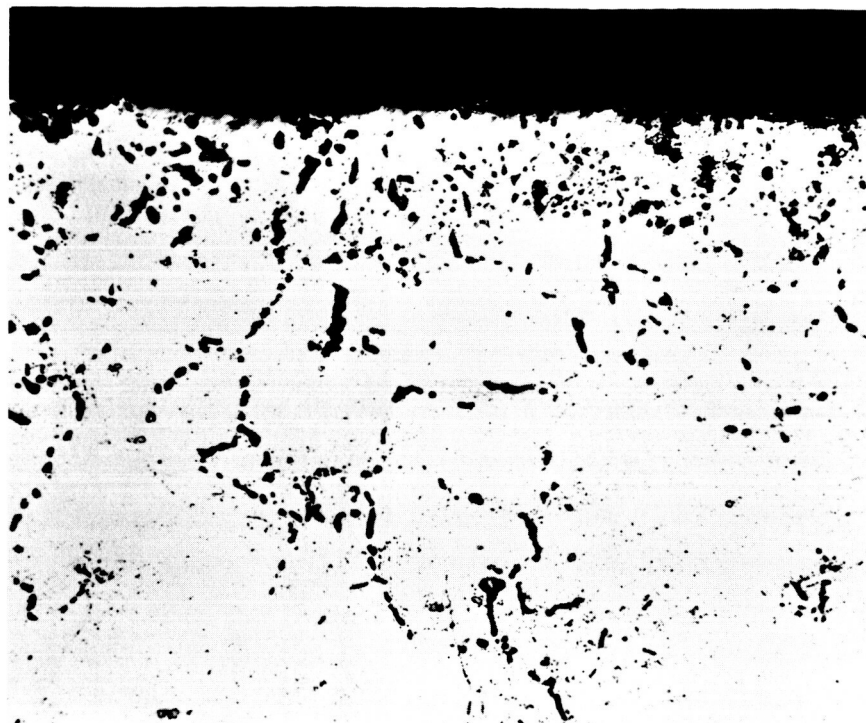
Examination of the specimens removed from capsule no. 5, which did not fail revealed corrosive attack in the form of grain boundary penetration and leaching. The depth of attack varied along the inside surface from less than 0.001 inch at the top to 0.005 inch near the bottom of the capsule. Figures 20 and 21 illustrate the extremes of attack incurred. As shown in Figure 20, the penetration incurred was less than .001 inch. However, as can be seen, there appears to be a second phase present within the grain boundaries to a depth of 0.009 inch. As of the present time the significance of this apparent second phase is not known. A microprobe analysis is being made.

In view of the extremely damaged condition of the capsules, the exact cause of failure could not be determined.

3.2.2 Results of Examination after the 1327 Hour Interruption

All four of the capsules were opened. Two of these still contained some LiF. These were capsule No. 2 (Rene 41) and capsule no. 10 (Udimet 500). From the examination of the radiographs, capsule no. 2 appeared to have a large amount of porosity in the bottom. Also, no indications of LiF could be found inside the capsule. However, once opened, the capsule appeared to be full of LiF. The capsule appeared to be good. Evidently, interpretation of the capsule radiograph was obscured by the extremely roughened condition of the capsule exterior.

Likewise, when capsule no. 10 was opened, it was found to still contain LiF. However, it also contained a fluid. Examination of the inside tube surface revealed several circumferential cracks in the wall. The cracks were found at the base and at the top of the longitudinal seam weld. The crack of the base of the weld apparently occurred during the fabrication of the capsule since a leak in that area was found during the filling of the capsule. This crack was sealed by welding and building up the area with filler material. Examination of this area did not show any indications of the crack penetrating the build up. The crack at the top of the capsule apparently occurred during test. Evidently the liquid found when the capsule was opened entered the capsule through this crack. Before the capsule was opened, the capsule surface was immersed in an ammonium oxalate solution, water, and Stoddard solvent. The purpose of immersing the capsule in the various liquids was to remove traces of lithium fluoride and dye penetrant from the outside capsule surface. Figures 22 and 23 show the general appearance of the cracks.



RDM 9504

250X

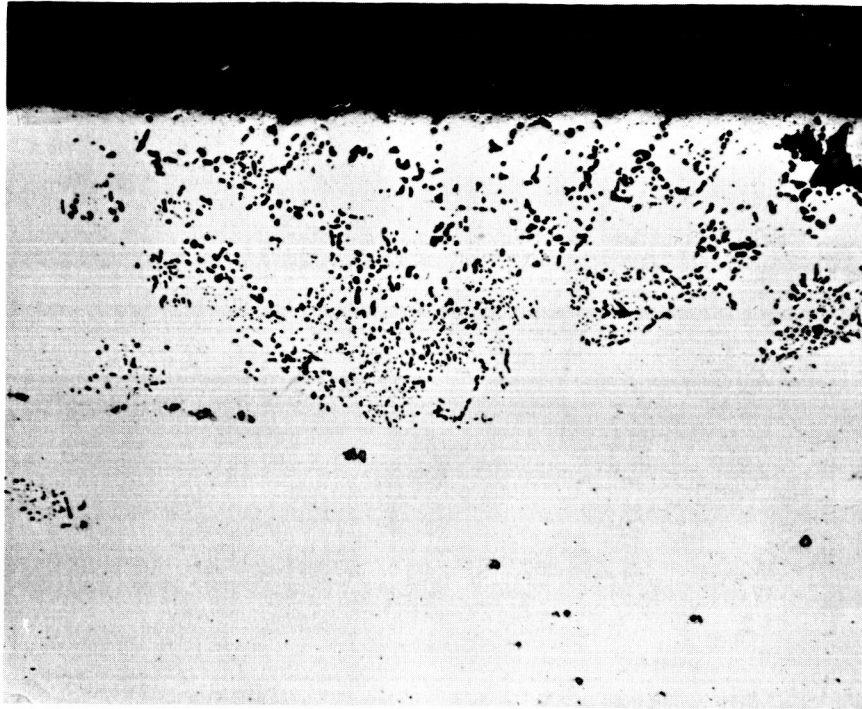
PHOTOMICROGRAPH OF SPECIMEN REMOVED FROM THE BOTTOM
OF RENE 41 CAPSULE NO. 8 SHOWING APPEARANCE AND SEVERITY
OF ATTACK. UNETCHED



RDM 9500

250X

PHOTOMICROGRAPH OF SPECIMEN REMOVED FROM THE TOP
OF RENE 41 CAPSULE NO. 5 SHOWING THE GENERAL APPEARANCE
OF THE INSIDE SURFACE. UNETCHED.

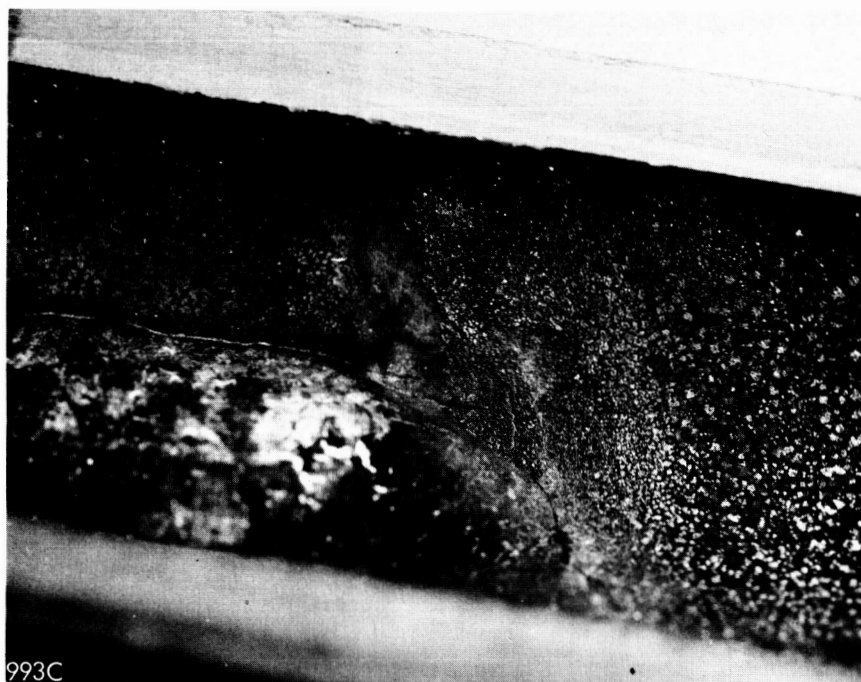


RDM 9498

250X

PHOTOMICROGRAPH OF SPECIMEN REMOVED FROM THE BOTTOM
OF RENE 41 CAPSULE NO. 5. SHOWING THE MAXIMUM AMOUNT OF
OF CORROSION INCURRED. UNETCHED

FIGURE 21



MACROGRAPH OF UDIMET 500 CAPSULE NO. 10 SHOWING THE CIRCUMFERENTIAL CRACK FOUND AT THE BOTTOM OF THE WELD BEAD. APPROXIMATELY 10 X (07686-1)



MACROGRAPH OF UDIMET 500 CAPSULE NO. 10 SHOWING
THE CIRCUMFERENTIAL CRACK FOUND AT THE TOP OF
THE WELD BEAD. APPROXIMATELY 10X (07686-3)

The two capsules, empty of LiF, were capsule no. 11 (Udimet 500) and capsule no. 18 (T. D. nickel). A circumferential crack was also found at the base of the weld in capsule no. 11. The crack is shown in Figure 24.

The results obtained through metallographic examination of specimens removed from the capsules are as follows and are summarized in Table II.

Rene 41, Capsule No. 2

Examination of the specimens removed from the capsule revealed a slight amount of attack in the form of grain boundary penetrations. The maximum depth of attack was .001 inch. This capsule had welded end caps only. And it was fabricated by boring a 3/8 inch hole through 1/2 inch Rene 41 bar stock. This capsule had not failed.

Udimet 500, Capsule No. 10

Metallographic examination of specimens removed at each crack revealed that both cracks had completely penetrated the tube wall. Also, as shown in Figure 25, around the cracks, attack in the form of leaching and intergranular penetration was found. The attack had completely penetrated the tube wall. Metallographic specimens removed from the same location, but from the opposite side of the tube where the crack was found also revealed a leaching and intergranular attack. However, as shown in Figure 26, the attack was limited to depths between .002 to .004 inches.

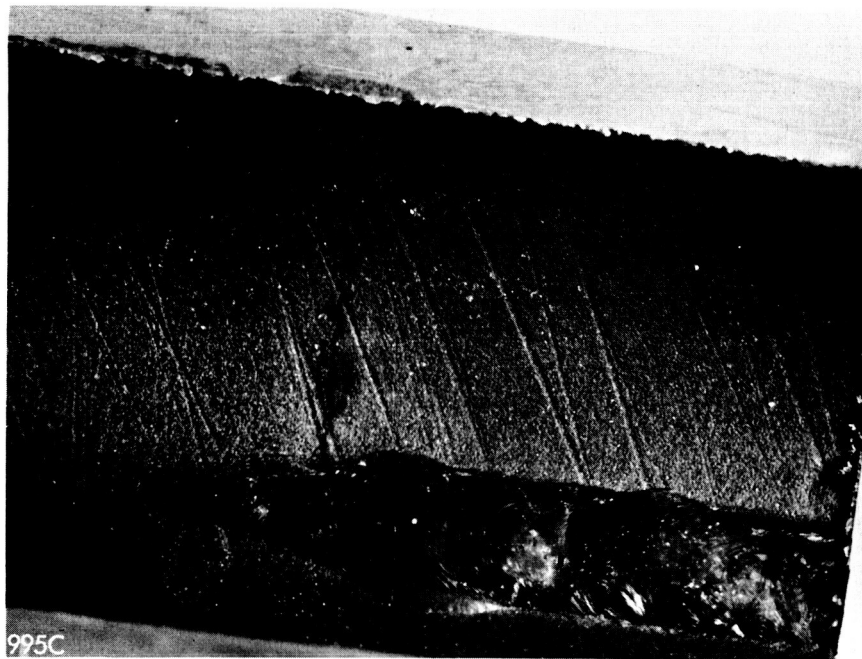
Udimet 500, Capsule No. 11

In general, the results found from metallographic examinations were the same as those reported for Capsule No. 10. However, only one crack, shown in Figure 24, was found in the tube wall. The condition of the tube wall around the crack was the same as reported for Capsule No. 10. The tube wall was completely penetrated. Away from the crack, leaching and intergranular penetration was found to a maximum depth of .003 inch.

Both Capsules 10 and 11 were fabricated from bar stock in the same manner as Rene 41 Capsule No. 2. However, besides the welded end caps, both capsules had longitudinal seam welds. The filler material used for the welds was Rene 41. Also, both capsules were tested in the as-welded condition. No post weld heat treatments were used.

T. D. Nickel, Capsule No. 18

As shown in Figure 27 a crack was found which penetrated the weld bead at the top of the capsule. Around the weld bead, attack in the form of intergranular penetration and leaching was found. The maximum depth of attack incurred was 0.004 inch. Examination of the TD nickel tube wall revealed a small amount of attack. Sections from both above and below the liquid level revealed a solution type of attack. The maximum depth found was less than .001 inch.



MACROGRAPH OF UDIMET 500 CAPSULE NO. 11 SHOWING
THE CIRCUMFERENTIAL CRACK FOUND AT THE TOP OF
THE WELD BEAD. APPROXIMATELY 10X (07686-2).

FIGURE 24



Outside Diameter

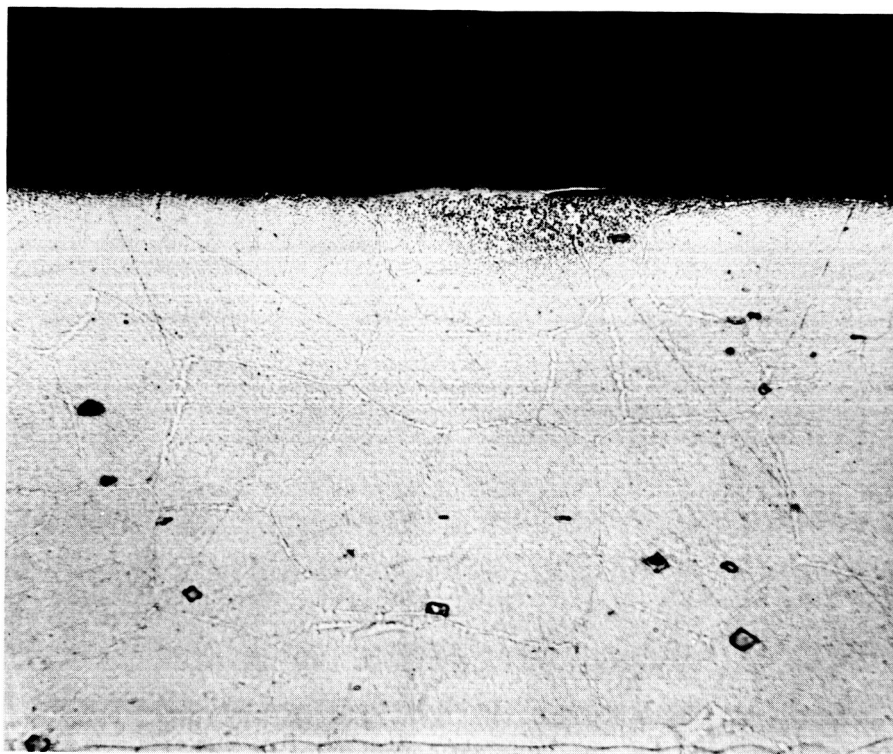
Inside Diameter

RDM 9998

50X

PHOTOMICROGRAPH OF UDIMET 500 CAPSULE NO. 10 TUBE
WALL SHOWING THE ATTACK INCURRED ON THE CRACK
SURFACE. REFRACTORY METALS ETCH.

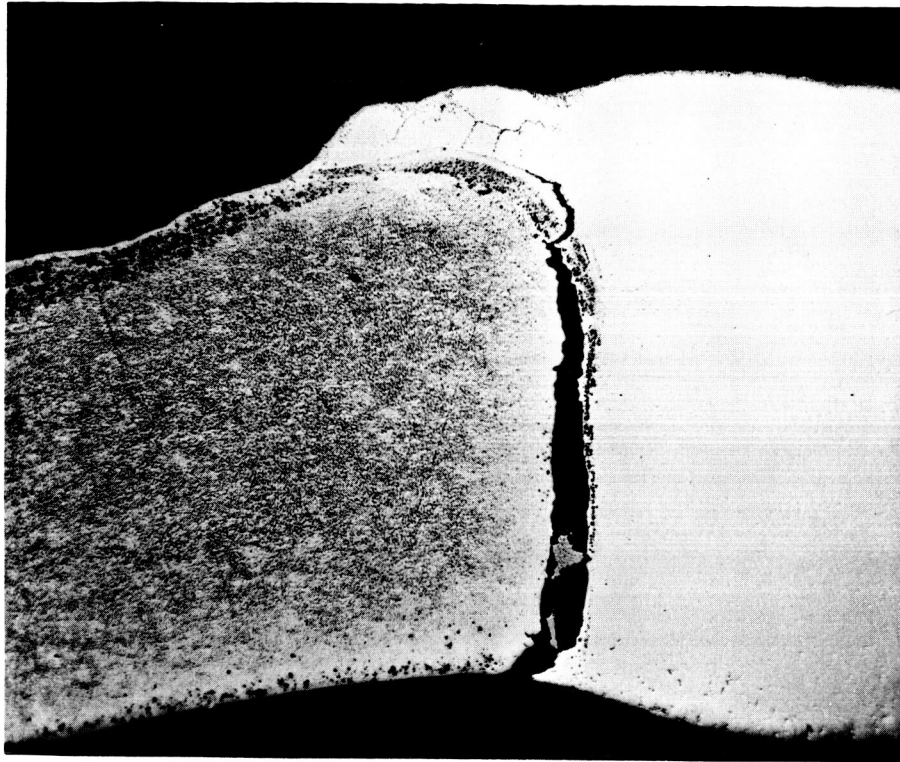
FIGURE 25



RDM 10,002 200X

PHOTOMICROGRAPH OF THE INSIDE SURFACE OF UDIMET 500
CAPSULE NO. 10 SHOWING THE MAXIMUM DEPTH OF ATTACK
INCURRED 100° AWAY FROM THE CRACK. REFRACTORY
METALS ETCH.

Rene 41



Outside Diameter

Inside Diameter

RDM 10,005 250X

PHOTOMICROGRAPH OF THE WELD BEAD IN TD NICKEL CAPSULE
NO. 18 SHOWING THE APPEARANCE OF THE CRACK AND CORROSION.
CARAPELLAS ETCH.

FIGURE 27

Capsule No. 18 was likewise fabricated in a manner similar to Rene 41 Capsule No. 2. Although it also had the longitudinal seam welds Rene 41 was used as the filler material. No post weld heat treatments were made on the capsule.

From the appearance of the cracks and the attack incurred in the various specimens examined, all three capsules, Udimet 500 Capsules 10 and 11, and TD Nickel Capsule No. 18 apparently suffered mechanical failures. In view of the 0.003 to 0.004 inch depth of attack found away from the cracks in both Udimet 500 capsules, it appears that the complete corrosive penetration of the tube wall around the cracks occurred after the mechanical failure of the capsule. Once the crack had formed, the lithium fluoride inside the capsule was exposed to the argon atmosphere that was present in the capsule container. As the previous container failure demonstrated, enough water vapor is present in the argon to allow generation of hydrogen fluoride, which is extremely corrosive at the temperatures of operation.

Although the extent of attack incurred in TD nickel Capsule No. 18 was less than that found in the Udimet 500 Capsules, the results obtained from the examination lead to the same conclusion. That is the failure occurred through mechanical means.

3.2.3 Summary

As discussed, all of the failures have occurred in capsules either fabricated from nickel base precipitation hardening alloys, or welded using a nickel base precipitation hardening alloy as the filler material. Also, the mode of failure appears to be mechanical in nature. And the location of the failure has been either near or adjacent to a weld area.

A successful welding of precipitation hardening nickel base alloys such as Rene 41 or Udimet 500 is a difficult art. Primarily, the difficulties result from the presence of the precipitation hardening constituents, aluminum and titanium. During welding, part of the heat affected zone is heated high enough to undergo age hardening and thus the precipitation of a $\text{Ni}_3(\text{Al}, \text{Ti})$ compounds. The precipitation of this compound results in a volumetric change in the base metal which causes a distortion of the metallic lattice. As a result, mechanical stresses are created within the heat affected zone. Also, the age hardened metal exhibits a ductility which is significantly lower than that of non age-hardened material. Along with this, non-uniform heating and cooling during welding and shrinkage of the weld bead during cooling can add to the stress level of the heat affected zone, especially if the part being welded is restrained. Because of the low ductility and stresses imposed, the heat affected zone is quite susceptible to failure through cracking.

The only way to prevent this is to weld the alloy in the solution treated condition, and immediately after welding again anneal and place the aluminum and titanium back into solution. However, in some cases, post-weld heat treatment is difficult because of the restraint created by the weld joint. The tubular capsules with the longitudinal seam weld are examples. During welding, the tube is subject to tensile hoop

stresses as a result of thermal expansion. Also, because of the configuration, the weld area and heat affected zones are subjected to restraint.

Considering the fact that the capsules were to be operated at temperatures between 1500°F and 1850°F it was decided to test the capsules in the as-welded condition. This was done in that it was felt that because of the restrained condition of the weld the capsules had about the same chance of surviving the test conditions without cracking as they would with any possible post weld heat treatment. Also, considering the probable configurations of the heat receiver, it might be difficult to provide an even post-weld heat treatment on the receiver. Thus, considering the probable conditions of operation of the receiver, it would be advantageous in fabrication if the receiver did not require post weld heat treatment.

In that no post-weld heat treatments were made on the capsules, the results point to the formation of excessive stresses in or about the weld area resulting from welding. Thus, if nickel base precipitation hardening alloys are used for the heat receiver, the welding practice during all joining operations must be carefully controlled to prevent the formation of excessive post weld stresses. Also post weld stress relief heat treatment of the welded structure probably will be required.

3.2.4 Task Schedule

The Project Schedule for Task II is shown in Figure 28.

PROJECT SCHEDULE FOR TASK II, THE MATERIALS CORROSION INVESTIGATION

PROJECT TITLE: Lithium Fluoride Corrosion and Material Compatibility Investigations

DATE: 1 Nov. 1963	MONTH OF	JULY	AUG.	SEPT.	OCT.	NOV.	DEC.	JAN.	FEB.	MARCH	APRIL	MAY	JUNE	JULY	AUG.	SEPT.	OCT.	NOV.	DEC.
ITEM																			
1. Materials Procurement																			
2. Capsule Fabrication																			
3. Furnace Test of Capsules																			
4. Evaluation and Analysis																			
5. Topical Report																			
REPORTS																			
1. Monthly																			
2. Quarterly																			
3. Topical Report																			

4.0 CURRENT PROBLEM AREAS

The only problem area which exists at the present time is the problem of understanding the failures which have occurred in Task II. Effort will be directed toward this end during the coming quarter. At press time, the test is again underway and the post interruption examination of the removed capsules is in progress.

5.0 PLANNED DIRECTION OF EFFORT FOR THE NEXT QUARTER

During the next quarter, the effort will be limited to the following:

1. Continuation of the first 2500-hour furnace test.
2. Examination and analysis of the two TD nickel capsules removed after the third interruption.
3. Continuation of discussions with NASA on additional corrosion testing and on additional work requested for the design study.

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